Materials Characterization

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Cathodoluminescence (CL) system

FEI Nova NanoSEM (FEG source) with:
- EDAX Apollo silicon drift detector (TE cooled)
- Gatan MonoCL3+

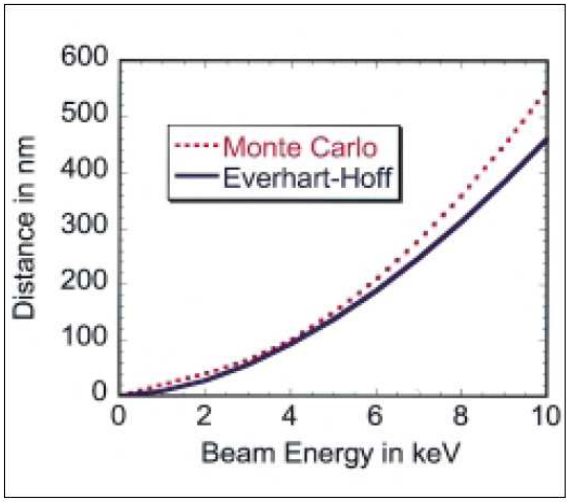
FEI SEM arrived Sept 1st and is installed.
- Landing energy 50 V to 30 kV, continuously adjustable
- Resolution High-vacuum: <1.0 nm @ 15 kV, 1.6 nm @ 1 kV

EDAX installed EDS detector on Sept. 15th. Boron lowest element detected.

Gatan to install detector on Sept. 22nd, MonoCL3+ detector allows for spectrum mapping with 150 line/mm grating or high resolution spectrum at a spot with a 1200 line/mm, grating. Short working distance parabolic mirror allows for higher resolution.

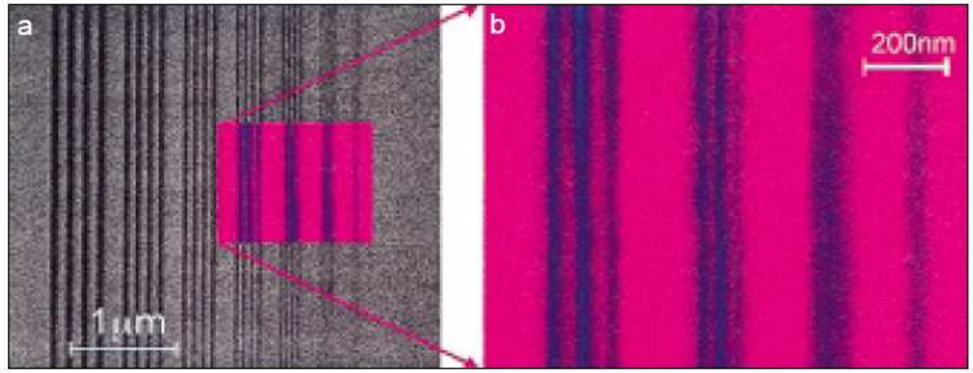
Tool and facility optimized for highest resolution possible.

Cathodoluminescence (CL) system

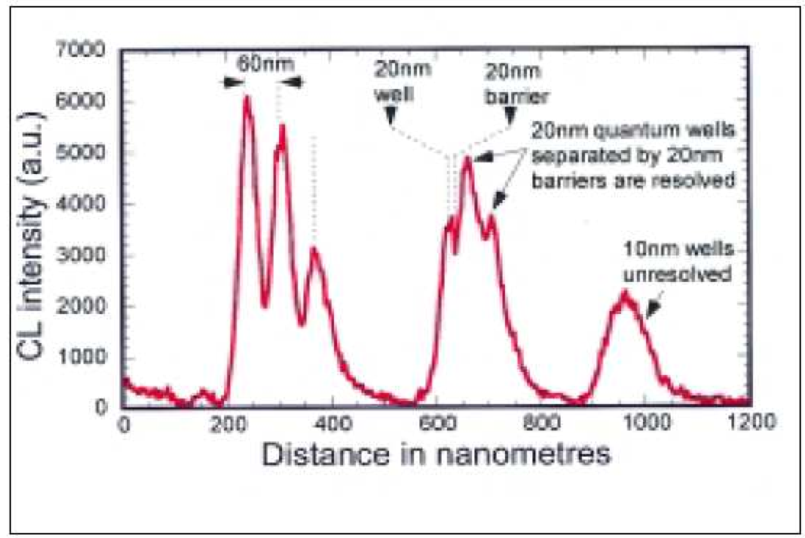


Electron penetration range simulated in GaAs

The FEI NOVA NanoSEM at the NRF is optimized for this type of CL spatial resolution, a cryo sample stage is required for this ultimate resolution.



GaAs/AlAs QW of 40nm, 30nm, 20nm, 10nm.



"Reaching the Spatial Resolution Limits of SEM-based CL and EBIC"
Carl E. Norman; MICROSCOPY AND ANALYSIS • MARCH 2002 (p9-12)

Review meeting Sept.2009

Passivation procedure and surfaces

Traditional passivation is PECVD SiNx, some in-situ CVD SiNx. Crystalline oxides show superior passivation, isolation and aging, but considered “novel”.

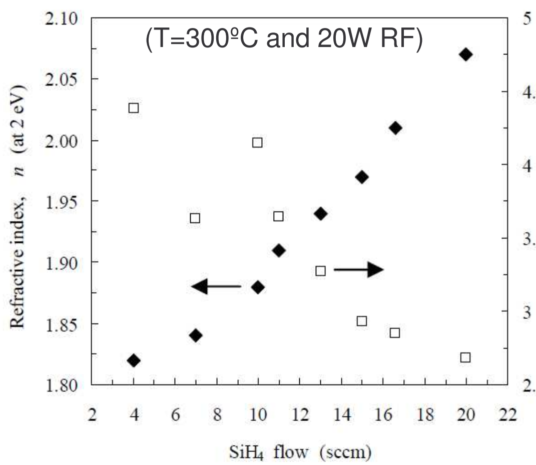
Passivation applied either in-situ post-epi, pre processing, or post processing.

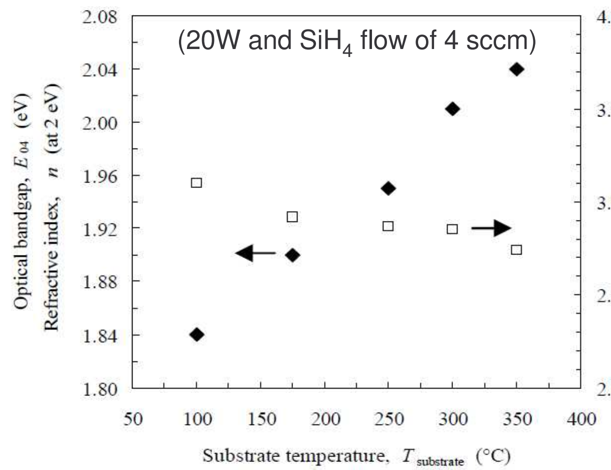
Various surface treatments: wet treatments of HF, buffered HF and NH_4OH, and dry treatments of ozone, N plasmas, NH3 anneals.

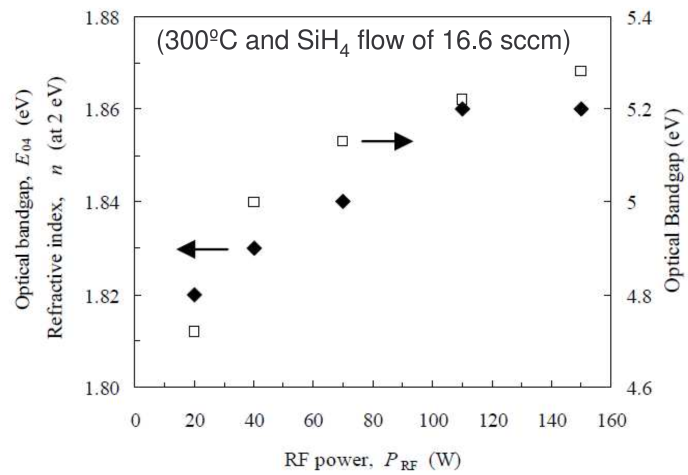
Three different surfaces: epi AlGaN, epi GaN, plasma etched GaN, and different crystal faces: c-plane and a-plane.

Typical passivation results in field passivation, but increases in drain leakage and gate leakage due to SiNx/III-Nitride interface.

Properties of PECVD SiNx







Refractive index ↑ with SiH_4 flow, RF power, substrate temperature.

Bandgap ↓ with SiH_4 flow and substrate temperature, ↑ with RF power.

Bandgap of >5eV with higher RF powers while the refractive index only shows a minor increase is an indication of densification of the SiNx films. (too high RF power damages the semiconductor surface)

SiNx layers deposited at a SiH4 flow >12 sccm contain Si-Si bonds reflecting the incorporation of amorphous-Si. (measurements of extinction coefficient *k*)

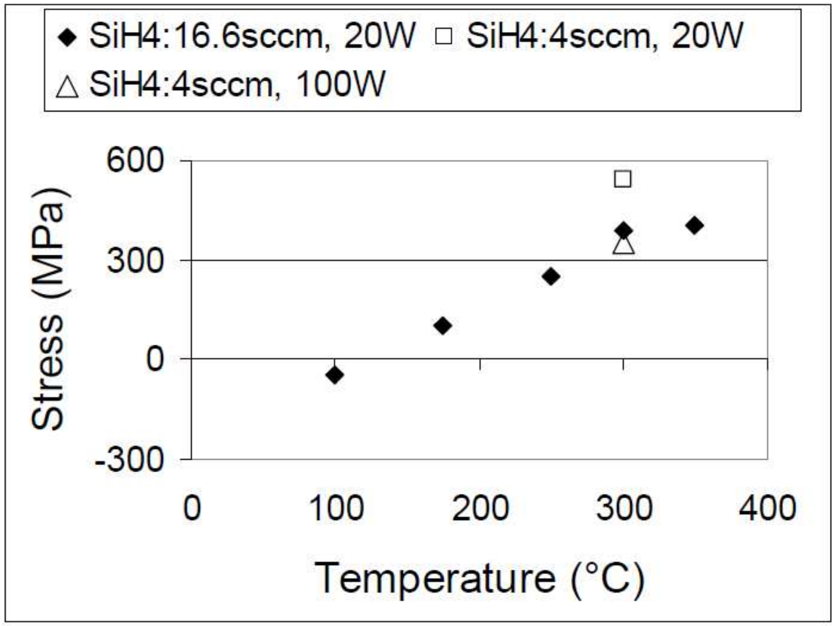
"Influence of the Structural and Compositional Properties of PECVD Silicon Nitride Layers on the Passivation of AlGaN/GaN HEMTs", F. Karouta et al.; ECS Transactions 16(7) p.181-191 (2008)

Properties of PECVD SiNx

Stress of SiNx layers

AlGaN barrier layers grown on top of GaN buffer layers in HEMT structures are tensile strained.

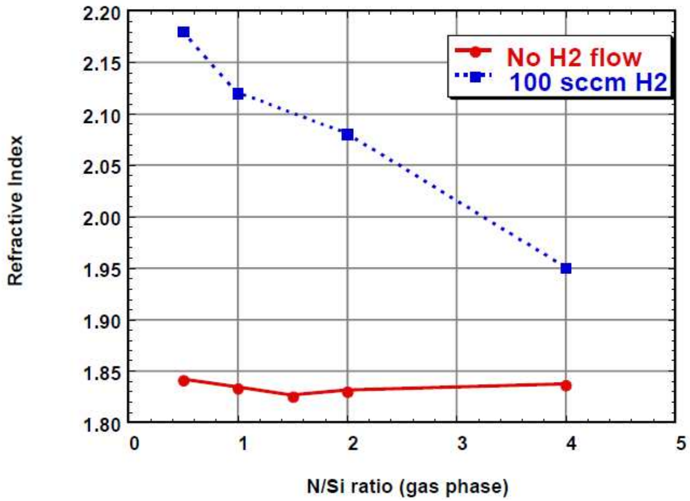
An increase in drain current is caused by an increase in sheet carrier density, which is caused by an increase in the piezoelectric polarization in the tensile strained AlGaN layer due to additional external tensile stress induced by the SiNx passivation film. (C. M. Jeon and J. L. Lee; *Appl. Phys. Lett.*, **86**, 172, 2005)



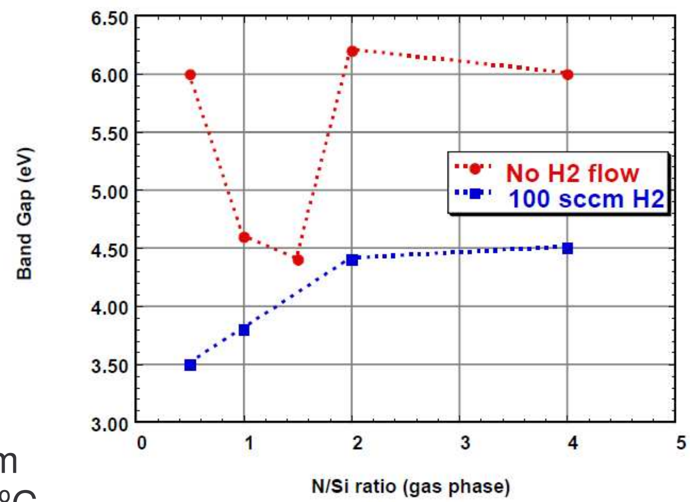
"Influence of the Structural and Compositional Properties of PECVD Silicon Nitride Layers on the Passivation of AlGaN/GaN HEMTs", F. Karouta et al.; ECS Transactions 16(7) p.181-191 (2008)

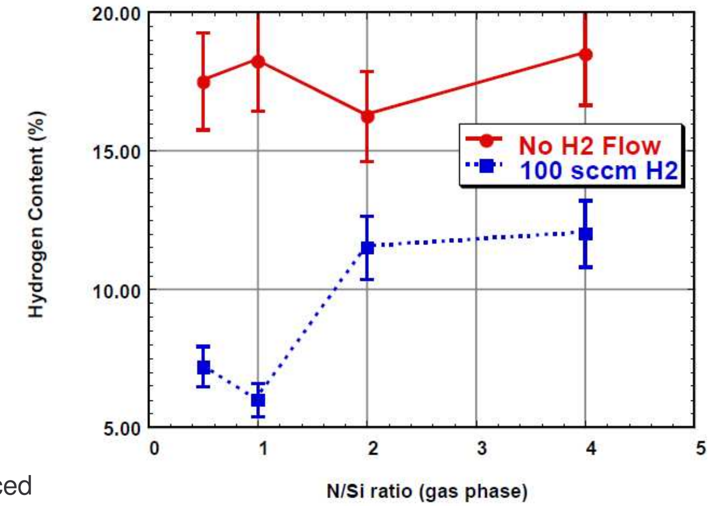
Properties of PECVD SiNx

Addition of hydrogen into the PECVD process offers another wide range of SiNx properties.



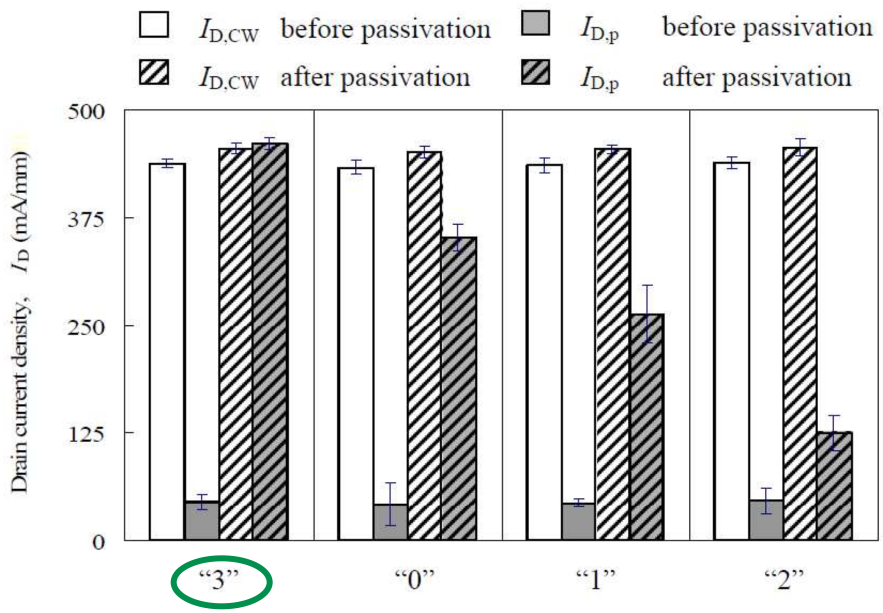
Prf = 1000 W
Silane = 10 sccm
Substrate = 380ºC

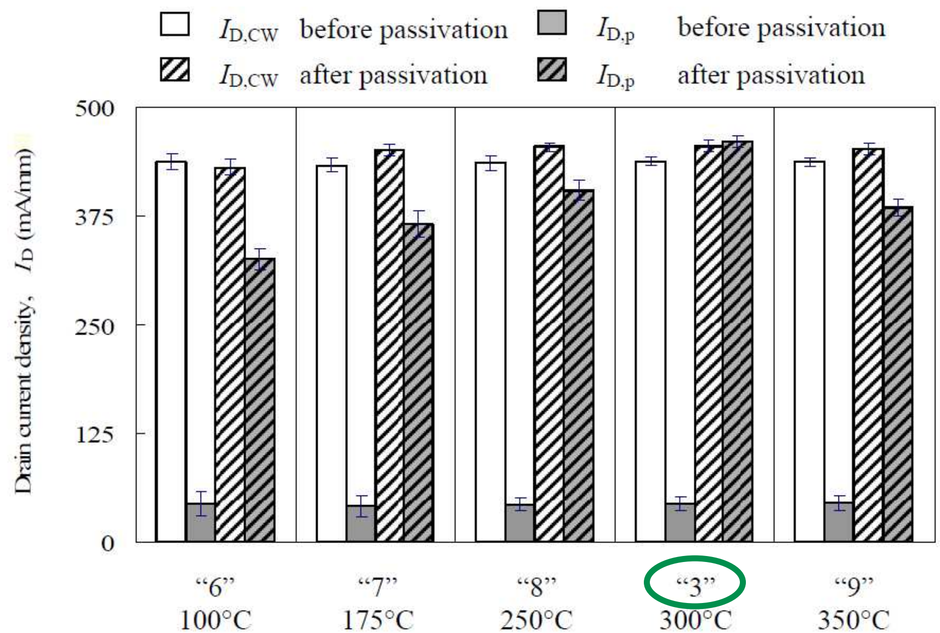




"Dielectric Properties of Silicon Nitride Deposited by High Density Plasma Enhanced Chemical Vapor Deposition"; J. Caughman et al.; 46th AVS National Symposium (1999)

Properties of PECVD SiNx and III-Nitride passivation





SiH_4=16.6sccm, P_{RF}=20W, p=650mTorr

Sample number	SiH_4 (sccm)	P_{RF} (W)	p (mTorr)	T (°C)
0	4	20	650	300
1	4	110	650	300
2	10	110	650	300
3	16.6	20	650	300

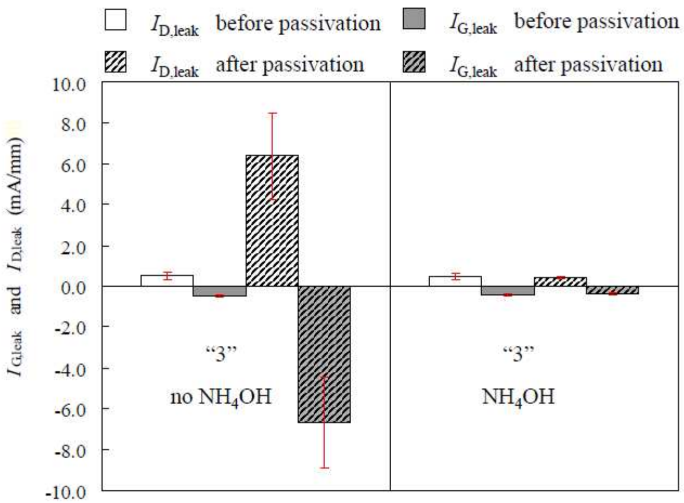
100nm SiNx on $Al_{0.25}Ga_{0.75}N$/GaN HEMT

Layer "3" did not result in the lowest gate and drain leakage, layers "0" and "2" showed the smallest leakage currents. Layer "1" resulted in the highest leakage currents.

Removal of SiNx passivation reduced gate leakage.

"Influence of the Structural and Compositional Properties of PECVD Silicon Nitride Layers on the Passivation of AlGaN/GaN HEMTs", F. Karouta et al.; ECS Transactions 16(7) p.181-191 (2008)

Pretreatment effects on passivation and leakage

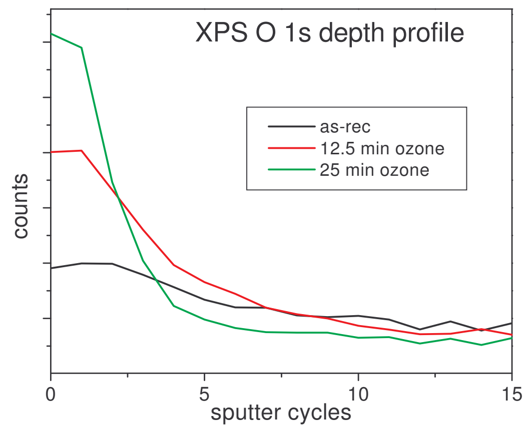


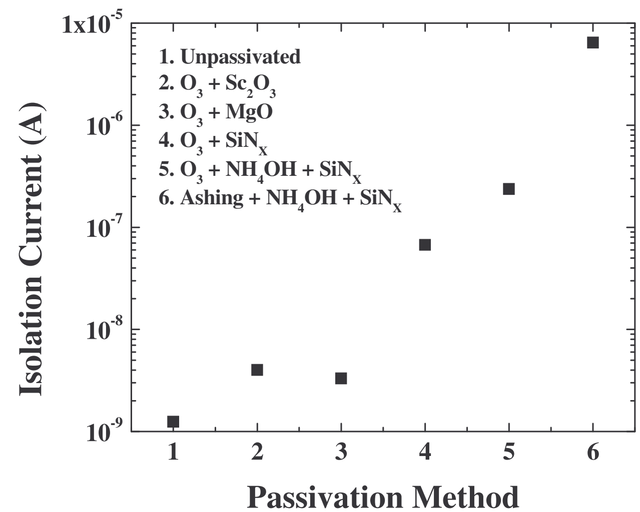
Influence of the NH_4OH dip on the gate and drain leakage current densities at pinch-off before and after passivation with 100 nm of film "3".

"Influence of the Structural and Compositional Properties of PECVD Silicon Nitride Layers on the Passivation of AlGaN/GaN HEMTs", F. Karouta et al.; ECS Transactions 16(7) p.181-191 (2008)

Oxygen surface conc. increases with ozone exposure time, however, oxygen depth does not. Surface converted to an oxynitride. (sample ~50℃)

Isolation region is plasma etched GaN.





"Novel Oxides and Reliability for the Passivation of AlGaN/GaN High Electron Mobility Transistor"
B.P. Gila et al.; presented *207th ECS, SOTAPOCS XLII, May 2005, Quebec*

Properties of plasma etched GaN

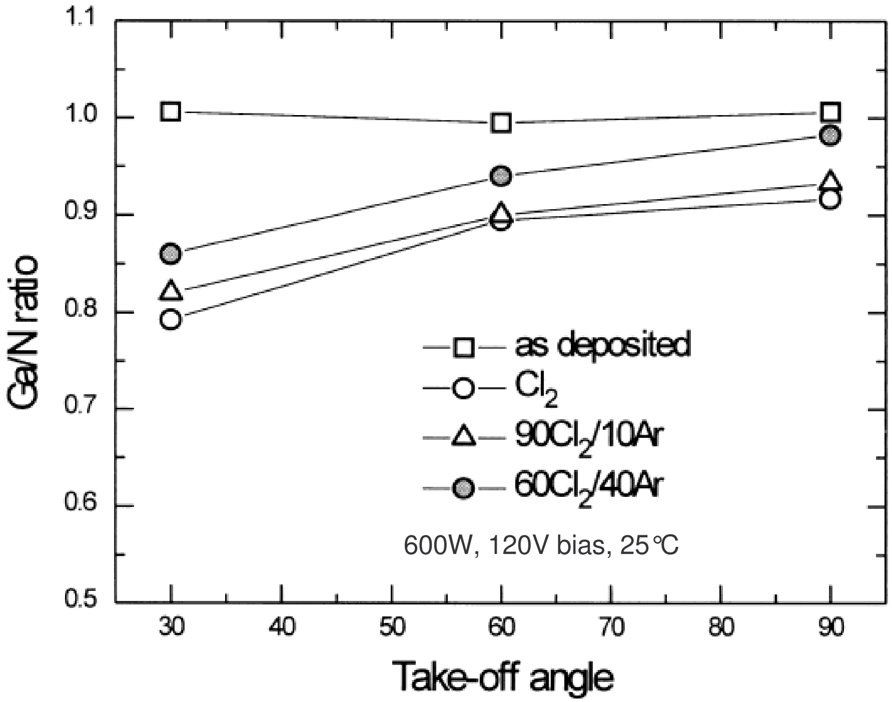
The increased chemical reaction between Cl and GaN can preferentially remove a component from the GaN surface during the etching.

Nitrogen-rich GaN is obtained and the GaN surface etched by 100% Cl2 shows the most nitrogen-rich surface.

The addition of Ar improved the stoichiometry of the surface, which shows the preferential removal of Ga from the GaN surface by the reaction of Cl and Ga in GaN under ion bombardment conditions enough to break GaN bonds.

In all cases, the RMS roughness of this surface is greater than the epi surface.

The deviation from stoichiometry will affect the passivation effectiveness and increase isolation leakage.



XPS measured Ga/N ratio vs. measurement angle (30° is near surface, 90° is 5-7nm sampling depth)

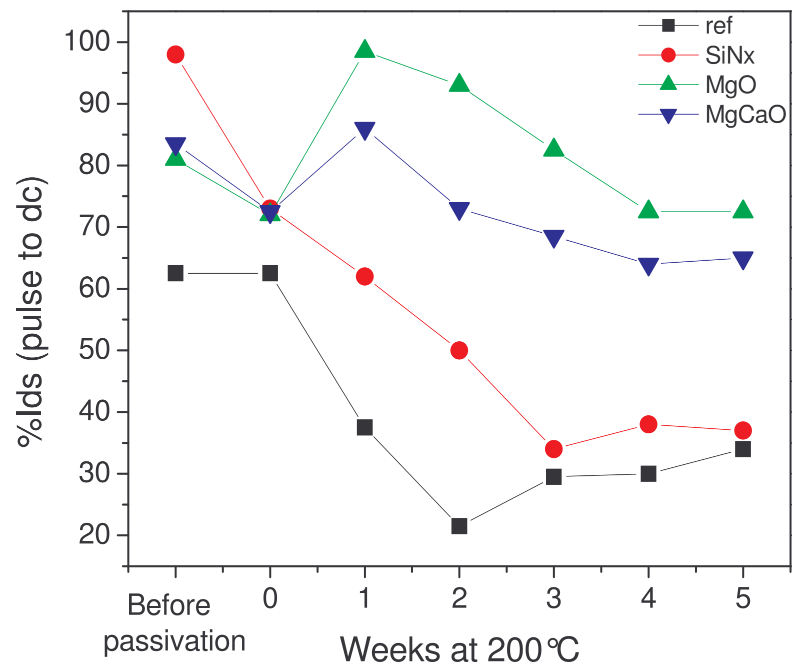
"A study of GaN etch mechanisms using inductively coupled Cl2/Ar plasmas"; Hyeon-Soo Kima et al.; Thin Solid Films 341 (1999) p.180-183

Thermal aging effects on passivation

Initial decrease due to UV/ozone exposure (traps emptied), 3 days to recover.

Crystalline oxides (type I heterostructure) show much higher passivation effect over SiNx (type II heterostructure) for III-Nitride FETs.

All passivation dielectrics are 10nm thick. Passivation effect limited to dielectric/semiconductor interface.



Surface passivation critical to performance and reliability

Chemical and thermal stability of passivation/semiconductor interface not well understood

Investigate relationship between surface band bending, interfacial chemistry and surface preparation as a function of temperature using XPS/UPS under controlled environment

XPS/UPS measurements

Design experiment to determine changes in SiNx passivation during device operation/stressing

- -MOCVD u-GaN/sapphire and AlGaN/GaN/sapphire
- -5nm PECVD SiNx deposited on substrates (250℃, 60W) to ensure the Ga peak is visible.

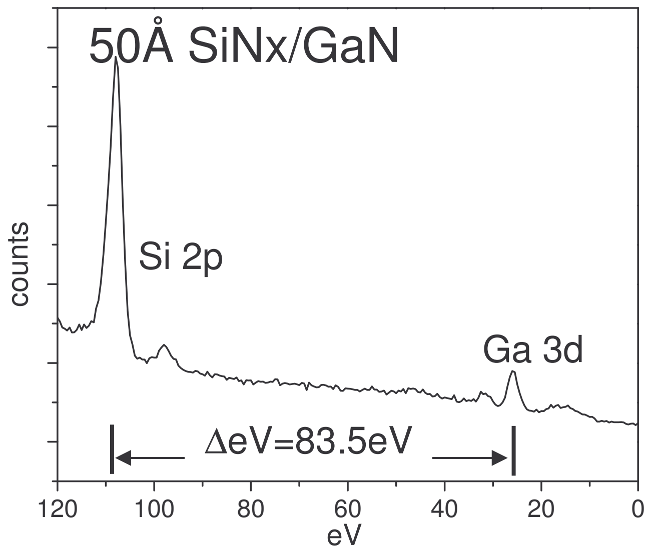
Use 200℃ anneal as a starting temperature and perform first anneals in the controlled environment of UHV. Temperature approximates device temperature under normal operation.

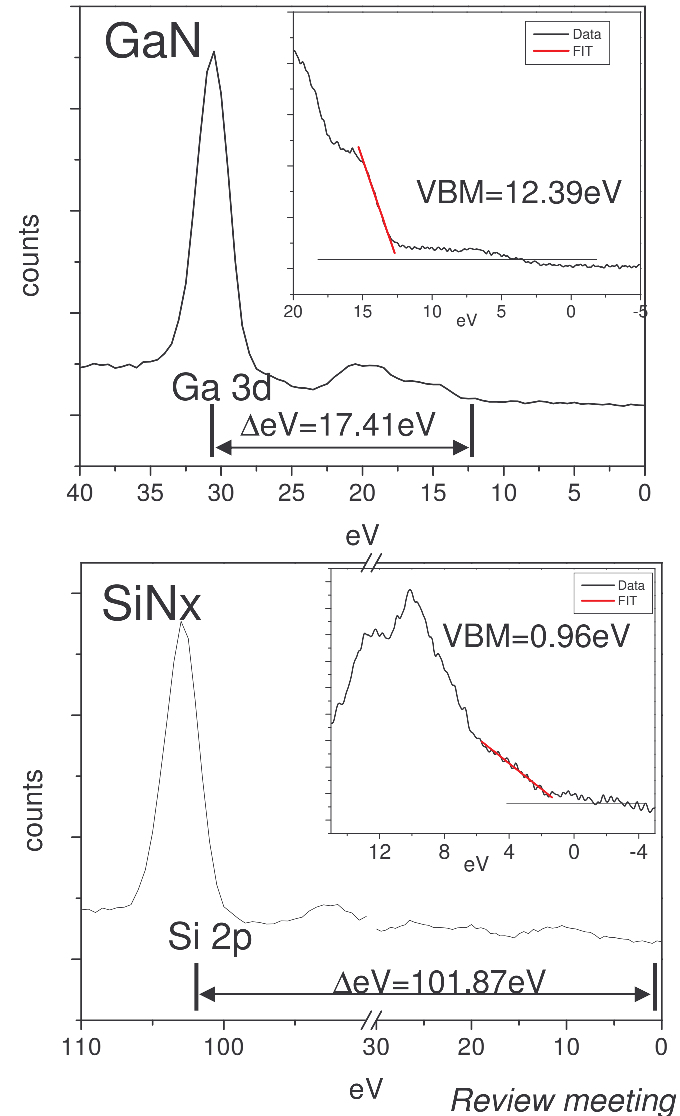
Calculate valance band offsets and look for changes in interface chemistry.

Follow-up experiments annealed in normal room atmosphere to determine ambient effects. (stressed die in open packages)

Quick review of Valance Band offset by XPS

Performing a VB offset measurement require 3 samples, 2 bulk and one combination sample.





For film A on substrate B;

$\Delta E_V = (E_V - E_{CL})_A - (E_V - E_{CL})_B + (\Delta E_{CL})_{A-B}$

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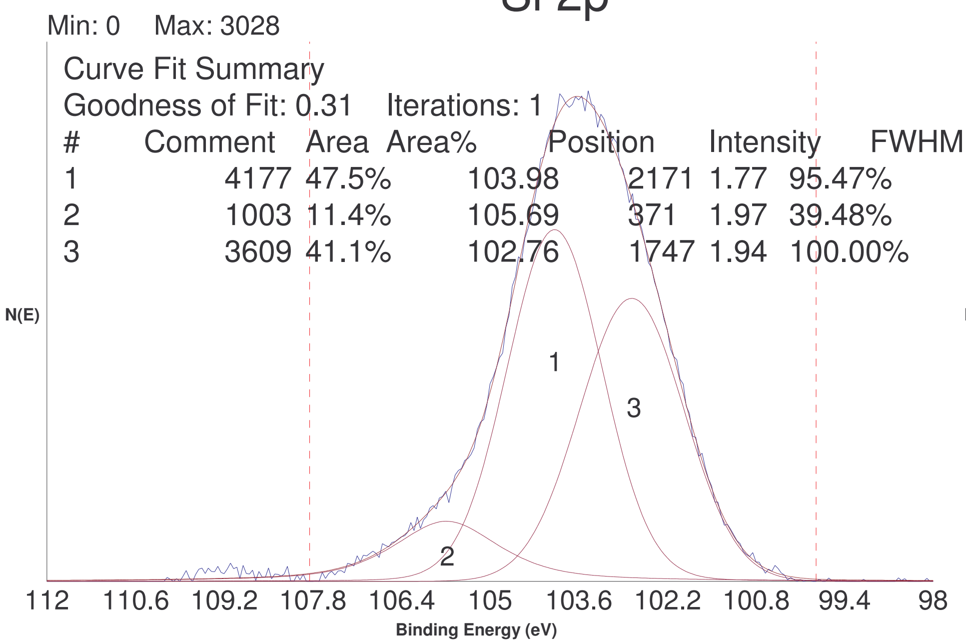
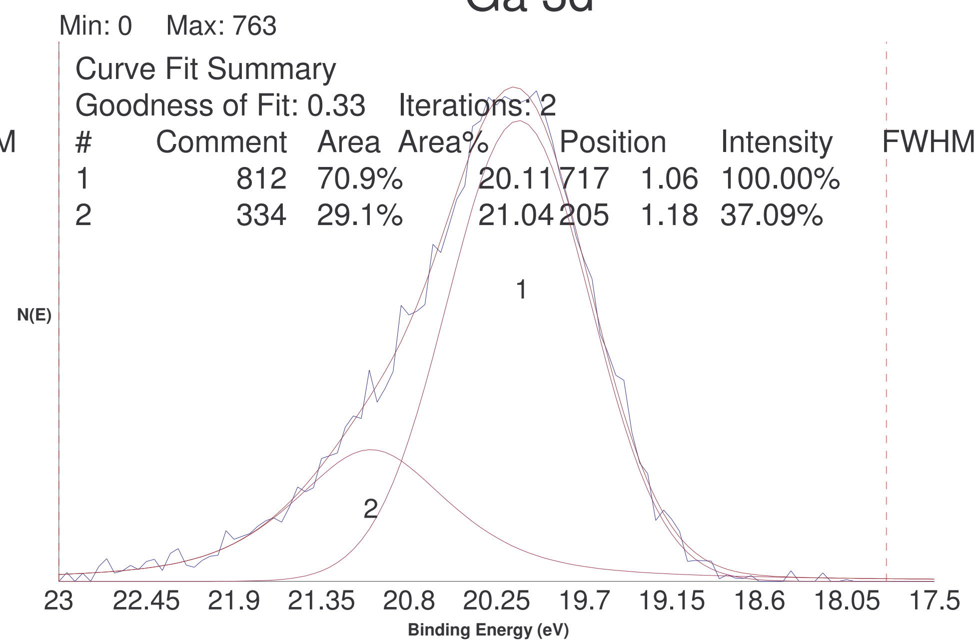
SiNx/u-GaN pre vacuum anneal

Si 2p



Ga 3d



Sample charging is inconsequential since peak separation is desired data and not peak position.

$$E_{CL}SiNx - E_{CL}GaN = 103.15 - 20.15 = 83.5\ eV$$

$$\Delta E_V = (E_V - E_{CL})_A - (E_V - E_{CL})_B + (\Delta E_{CL})_{A\text{-}B}$$

$$= (-101.87) - (-17.41) + (83.5)$$

$$= -0.96\ eV$$

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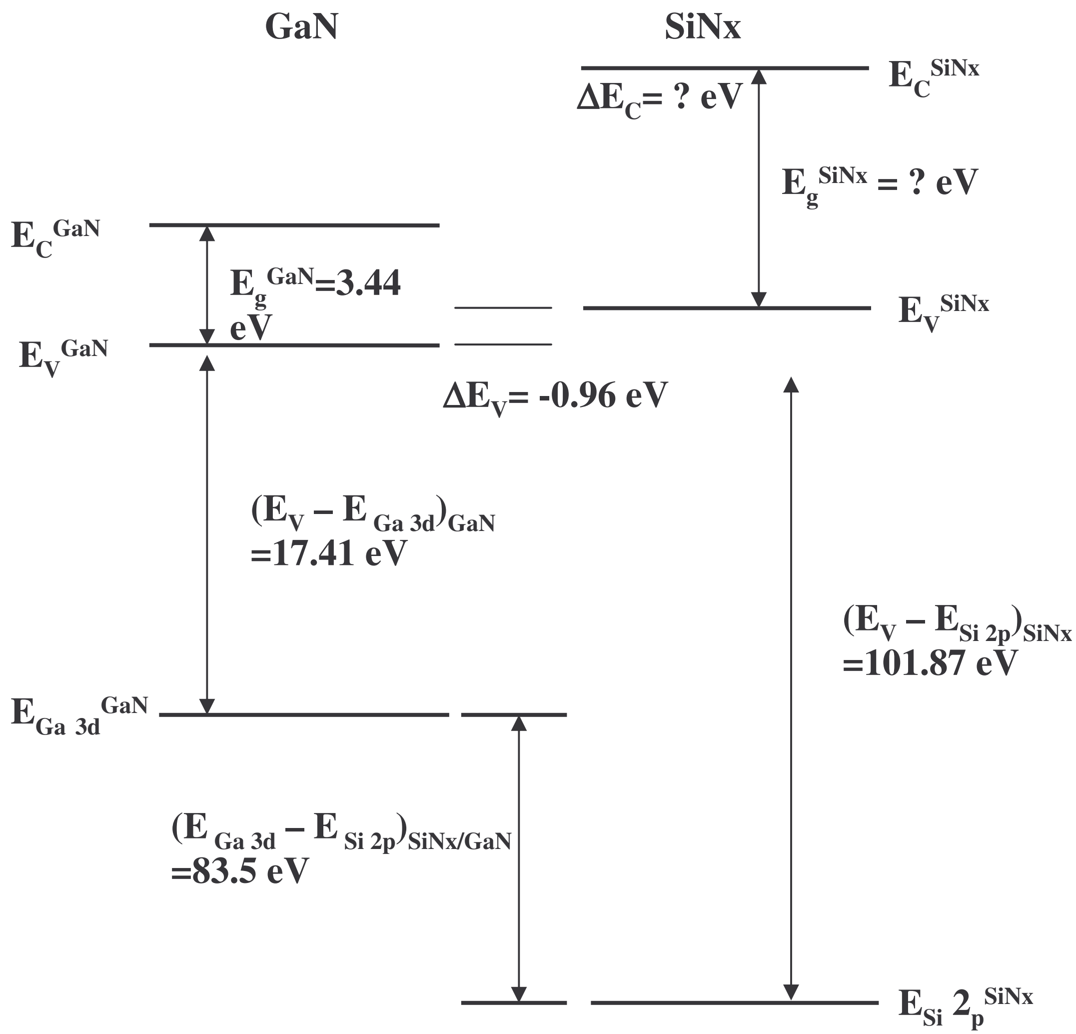
VB offset for pre-vacuum anneal SiNx on u-GaN



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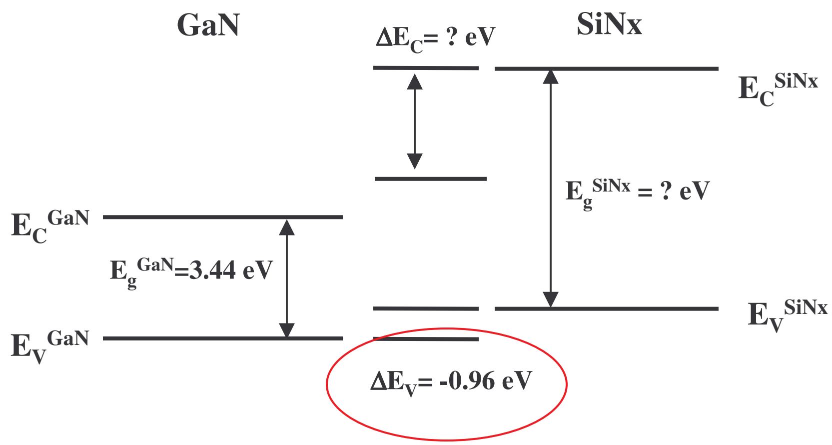
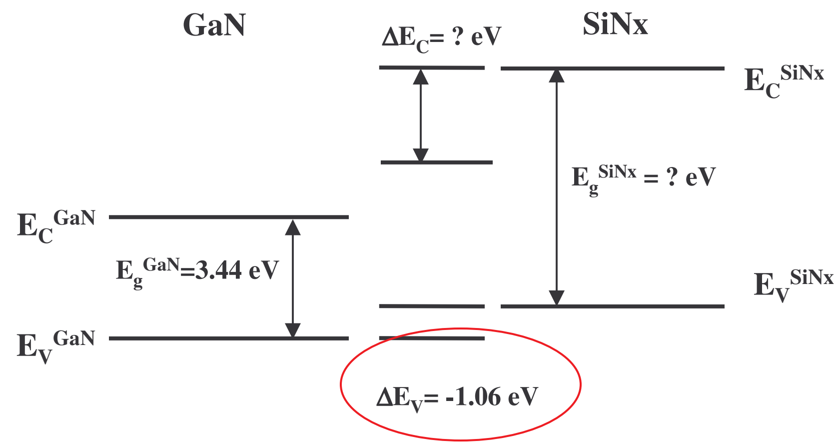
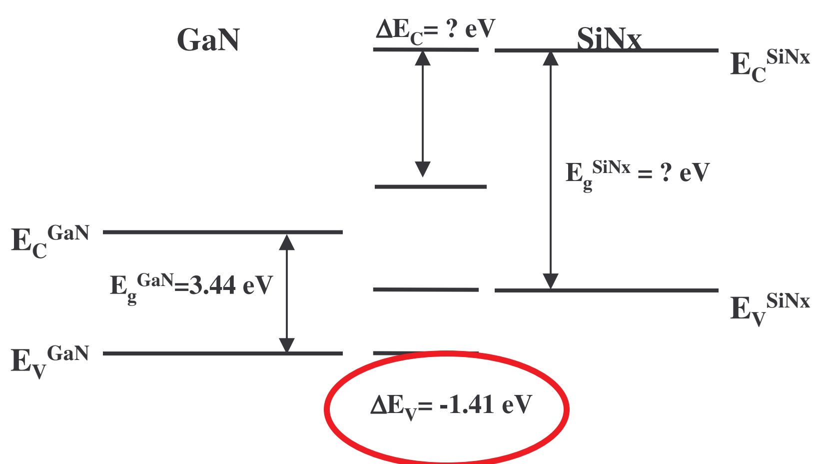
SiNx/u-GaN VB offset diagrams



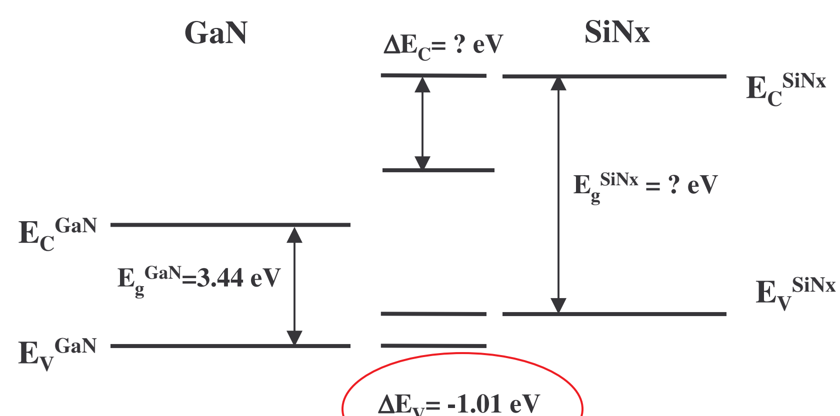
Pre vacuum anneal



During vacuum anneal (~200℃)



Post vacuum anneal



Post atmospheric anneal (~200℃)

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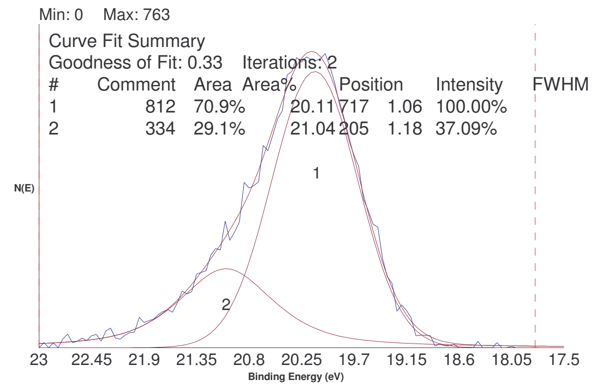
SiNx/u-GaN XPS peak evolution

Ga 3d peak shape with annealing and cooling.

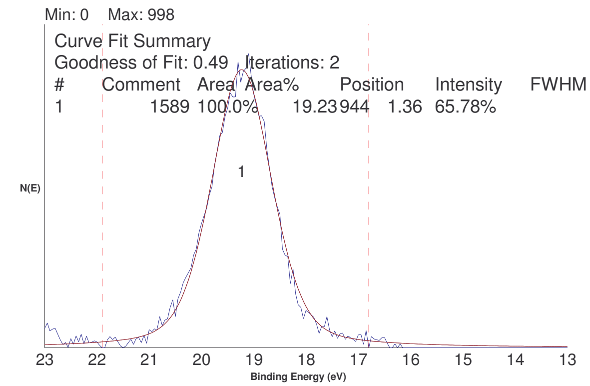
Presences of Ga-O bonding in pre vacuum anneal and post ex-situ anneal.

Si 2p peak shape not significantly changed during annealing and cooling.

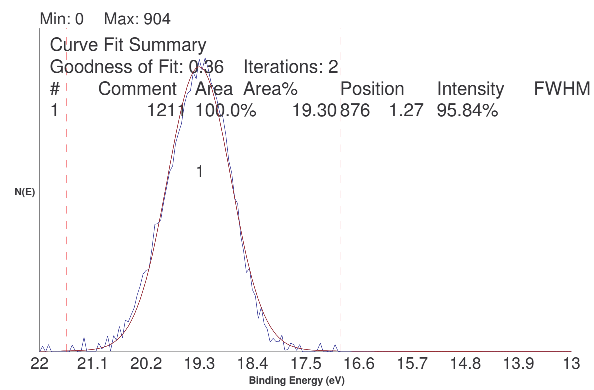
Is Ga-O responsible for ~0.4eV change in VB offset?



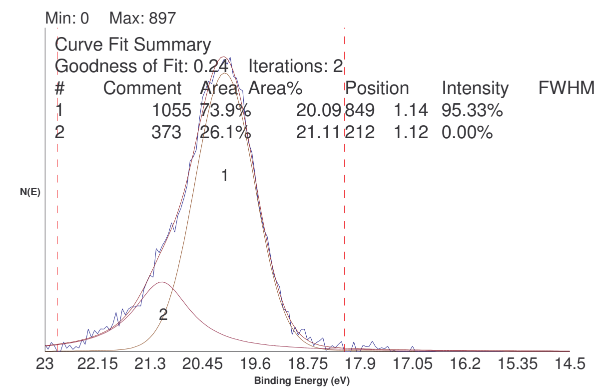
Pre vacuum anneal



Vacuum anneal 200℃



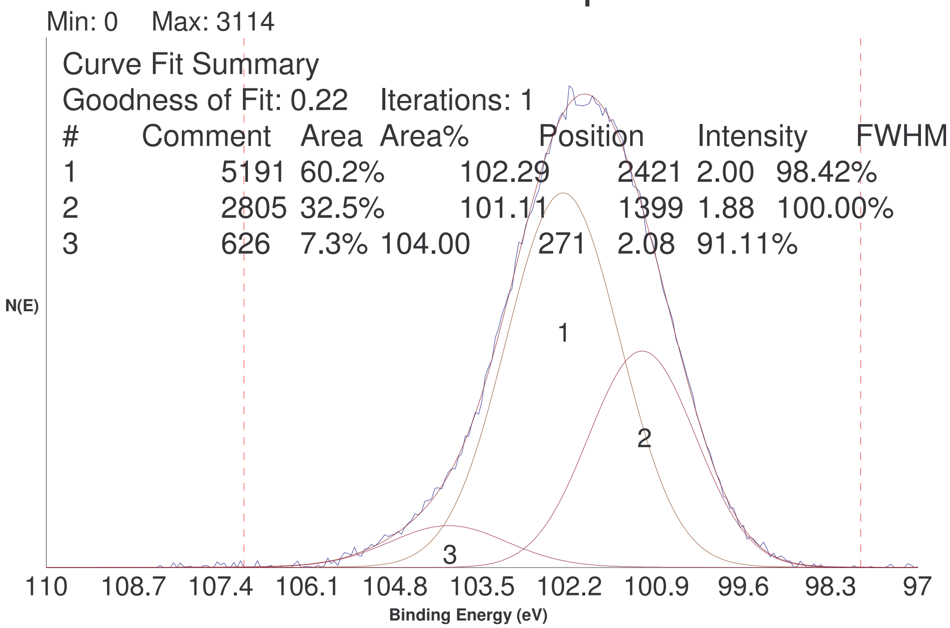
Post vacuum anneal



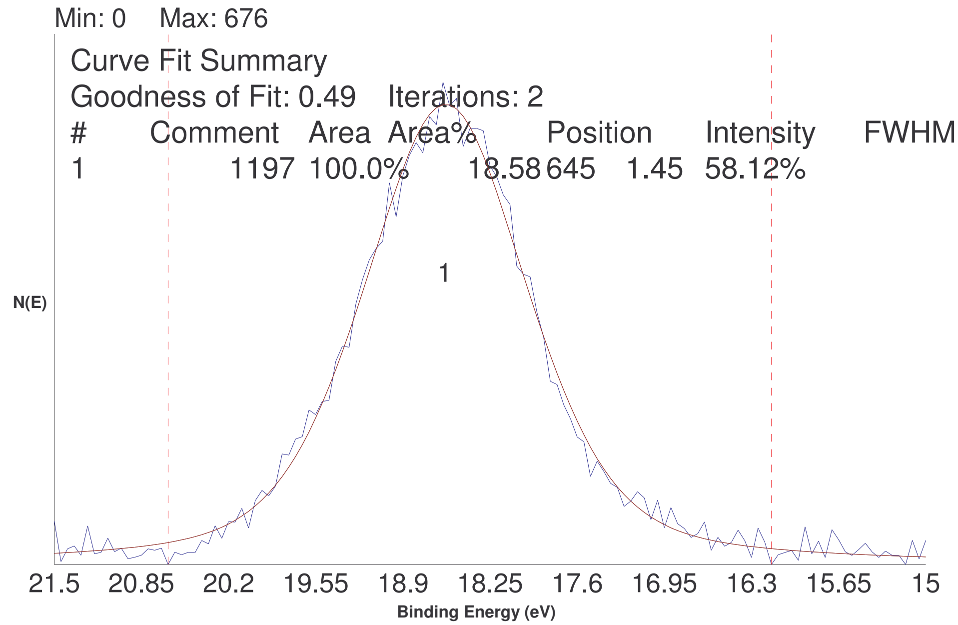
Post ex-situ 200℃ anneal

SiNx/AlGaN/GaN HEMT pre vacuum anneal

Si 2p



Ga 3d



$$E_{CL}SiNx - E_{CL}GaN = 101.95 - 18.58 = 83.37 \text{ eV}$$

$$\Delta E_V = (E_V - E_{CL})_A - (E_V - E_{CL})_B + (\Delta E_{CL})_{A\text{-}B}$$
$$= (-101.87) - (-17.48) + (83.37)$$
$$= -1.06 \text{ eV}$$

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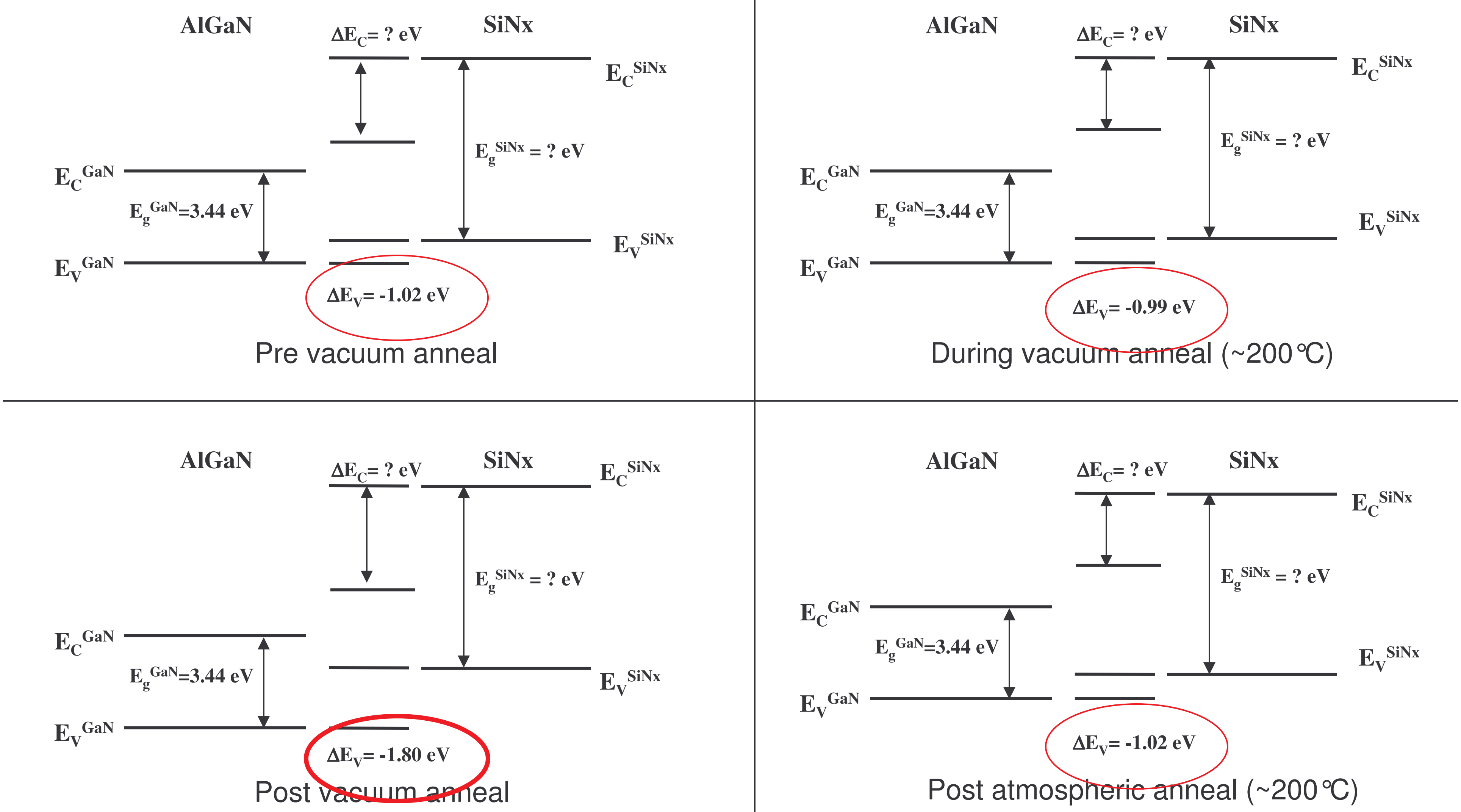
SiNx/AlGaN/GaN HEMT VB offset diagrams



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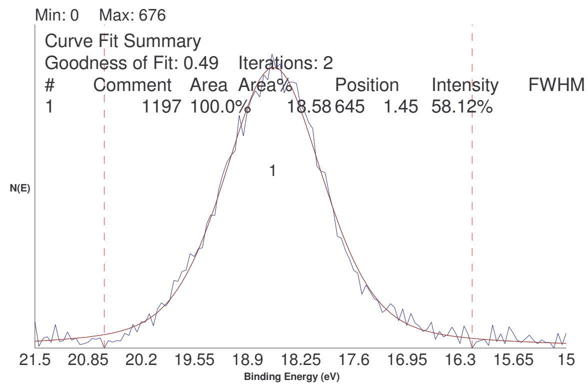
SiNx/AlGaN/GaN HEMT XPS peak evolution

Ga 3d peak shape with annealing and cooling.

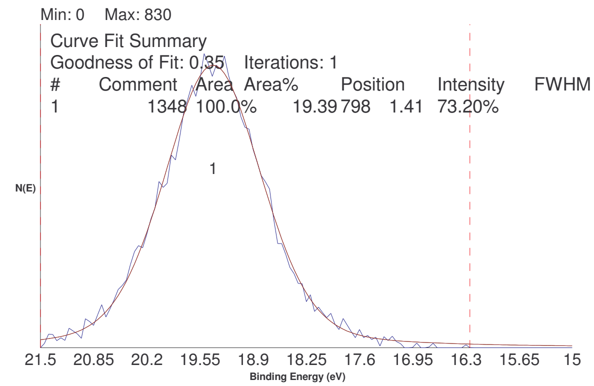
No presences of Ga-O bonding in spectra, only Ga-N bonding type determined.

Si 2p peak shape not significantly changed during annealing and cooling.

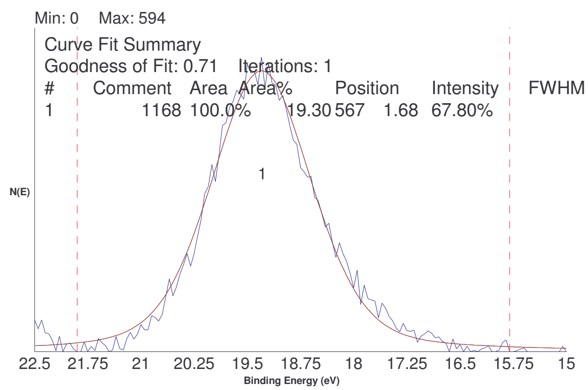
Ga-O cannot be responsible for ~0.8eV change in VB offset.



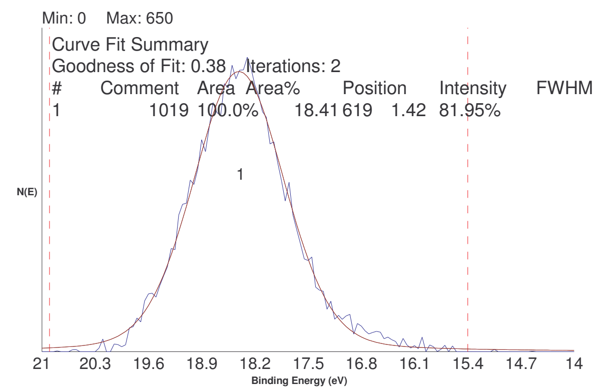
Pre vacuum anneal



Vacuum anneal 200℃



Post vacuum anneal



Post ex-situ 200℃ anneal

u-GaN valance band and core level measurements

Ec-Ev (GaN) = 20.30 – 2.905 = 17.40 eV
pre vacuum anneal

Ec-Ev (GaN) = 19.05 – 1.285 = 17.765 eV
at 200℃ in UHV

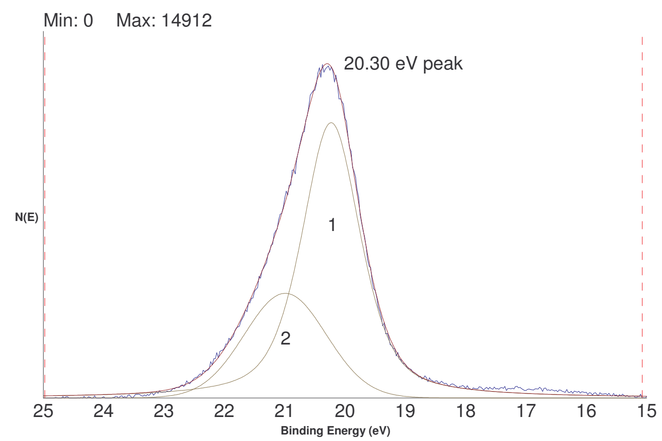
Ec-Ev (GaN) = 19.35 – 1.944 = 17.406 eV
post vacuum anneal

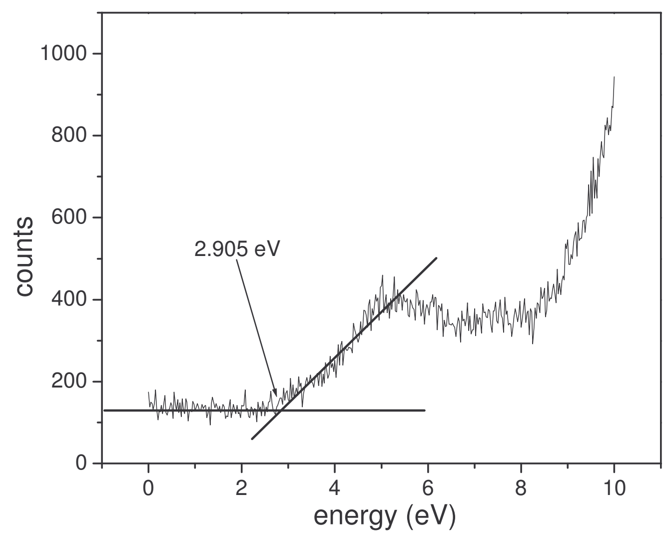
Ec-Ev (GaN) = 19.5 – 2.081 = 17.419 eV
post vacuum anneal (+1 day)

u-GaN without SiNx layer shows a shift in VBM only while at 200℃.

Ga-O portion of peak is consistent throughout the anneal.

VBM recovers to pre anneal value and is stable.





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Summary

SiNx on u-GaN

- Shift in VB of -0.4eV after vacuum anneal at 200℃. Ga 3d peak lost Ga-O portion. No significant changes in Si 2p peak.
- VB offset recovers to initial value after anneal in air at 200℃. Ga 3d peak recovered Ga-O portion. Peak positions and relative intensities are nearly identical to pre vacuum anneal values.

SiNx on HEMT

- Shift in VBM of -0.9eV after vacuum anneal. No determined Ga-O bonding in the Ga 3d peak.
- VB offset recovers to initial value after air anneal, similar to SiNx/u-GaN sample.

u-GaN

- Shift in Core Level to Valance Band only at anneal temperature. Returns to pre anneal value upon cool down. Ga 3d peak shape consistent throughout anneal, no loss of Ga-O portion.

Different mechanism for the two different samples due to the significant difference in VB change.